



Year: 2019

Effect of temporary cement removal methods from human dentin on zirconia-dentin adhesion

Januário, Ana Beatriz do Nascimento ; Moura, Dayanne Monielle Duarte ; de Araújo, Arthur Magno Medeiros ; Dal Piva, Amanda Maria de Oliveira ; Özcan, Mutlu ; Bottino, Marco Antonio ; Souza, Rodrigo Othávio Assunção

Abstract: This study evaluated the effect of temporary cement residue removal methods from human coronary dentin on the bond strength of adhesively-luted zirconia on dentin. Forty non-cariou human molars were embedded in acrylic resin and the dentin surfaces were exposed. Temporary acrylic crowns were provisionally cemented with zinc oxide cement without eugenol and stored in distilled water (37 °C/15 days). After crown removal, the excess temporary cement was removed from dentin according to one of the following cleaning methods: (n = 8 per group): (a) air-water rinse (AW), (b) pumice paste (PP), (c) air-abrasion with aluminum oxide particles (Al₂O₃) (AA), (d) sodium bicarbonate spray (SB) or (e) glycine powder (CP). Forty zirconia cylinders were made and each cylinder was adhesively luted onto each tooth after adhesive resin (Scotch Bond Universal, 3 M ESPE-SBU) application using resin cement (RelyX Ultimate, 3 M ESPE) and photo-polymerized from each surface for 20 s. The bonded specimens were stored in distilled water (37 °C) for 90 days. The bonded interface was loaded under shear (1 mm/min). Data (MPa) were analyzed using 1-way ANOVA and Tukey's test ($\alpha = 0.05$). Mean bond strength was significantly affected by the cleaning method ($p = 0.0289$). Cleaning with AA method resulted in significantly higher bond strength than with SB ($p < 0.05$) but similar to CP, PP and AW ($p > 0.05$). All cleaning methods were effective in removing temporary resin cement from dentin surfaces. Air-abrasion with aluminum oxide particles was more effective than with sodium bicarbonate spray promoting adhesion between zirconia and dentin.

DOI: <https://doi.org/10.1080/01694243.2019.1630163>

Posted at the Zurich Open Repository and Archive, University of Zurich

ZORA URL: <https://doi.org/10.5167/uzh-183930>

Journal Article

Accepted Version

Originally published at:

Januário, Ana Beatriz do Nascimento; Moura, Dayanne Monielle Duarte; de Araújo, Arthur Magno Medeiros; Dal Piva, Amanda Maria de Oliveira; Özcan, Mutlu; Bottino, Marco Antonio; Souza, Rodrigo Othávio Assunção (2019). Effect of temporary cement removal methods from human dentin on zirconia-dentin adhesion. *Journal of Adhesion Science and Technology*, 33(19):2112-2127.

DOI: <https://doi.org/10.1080/01694243.2019.1630163>

Effect of temporary cement removal methods from human dentin on zirconia-dentin adhesion

Ana Beatriz do Nascimento Januário, DDS^a / Dayanne Monielle Duarte Moura, DDS, PhD^b / Arthur Magno Medeiros de Araújo, DDS^c / Amanda Maria de Oliveira Dal Piva, DDS, PhD^d / Mutlu Özcan, Dr.med.dent., Ph.D^e / Marco Antonio Bottino, DDS, PhD^f / Rodrigo Othávio Assunção Souza, DDS, PhD^g

^aDDS, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Natal, Brazil

^bDDS, PhD student, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Natal, Brazil

^cDDS, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Natal, Brazil

^dDDS, PhD student, São Paulo State University (UNESP), Institute of Science and Technology, São José dos Campos, Brazil

^eProfessor, University of Zurich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland

^fProfessor, São Paulo State University (UNESP), Institute of Science and Technology, São José dos Campos, Brazil

^gProfessor, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Natal, Brazil

Short title: *Effect of temporary resin cleaning from dentin on adhesion*

***Part of this study has been presented at the Annual Meeting of Academy of Dental Materials in Porto de Galinhas, Pernambuco/Brazil, 2018, as a poster presentation.**

Correspondance to: *Adjunct Professor Rodrigo O. A. Souza, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Av. Salgado Filho, 1787, Lagoa Nova, Natal/RN, CEP: 59056-000, Brasil. e-mail: rodrigoothavio@gmail.com*

Abstract: This study evaluated the effect of temporary cement residue removal methods from human coronary dentin on the bond strength of adhesively-luted zirconia on dentin. Forty non-carious human molars were embedded in acrylic resin and the dentin surfaces were exposed. Temporary acrylic crowns were provisionally cemented with zinc oxide cement without eugenol and stored in distilled water (37°C/15 days). After crown removal, the excess temporary cement was removed from dentin according to one of the following cleaning methods: (n=8 per group): a) air-water rinse (AW), b) pumice paste (PP), c) air-abrasion with aluminum oxide particles (Al_2O_3) (AA), d) sodium bicarbonate spray (SB) or e) glycine powder (CP). Forty zirconia cylinders were made and each cylinder was adhesively luted onto each tooth after adhesive resin (Scotch Bond Universal, 3M ESPE-SBU) application using resin cement (RelyX Ultimate, 3M ESPE) and photo-polymerized from each surface for 20 s. The bonded specimens were stored in distilled water (37°C) for 90 days. The bonded interface was loaded under shear (1 mm/min). Data (MPa) were analyzed using 1-way ANOVA and Tukey's test ($\alpha=0.05$). Mean bond strength was significantly affected by the cleaning method ($p=0.0289$). Cleaning with AA method resulted in significantly higher bond strength than with SB ($p<0.05$) but similar to CP, PP and AW ($p>0.05$). All cleaning methods were effective in removing temporary resin cement from dentin surfaces. Air-abrasion with aluminum oxide particles was more effective than with sodium bicarbonate spray promoting adhesion between zirconia and dentin.

Keywords: Adhesion; Cleaning methods; Dentin; Resin cements; Shear bond strength; Zirconia

Introduction

In most cases, indirect restorations are performed in multiple clinical sessions that necessitates the provisional phase using temporary restorations that is essential for patient comfort, social interactions, maintaining periodontal health and the occlusal relationships during planning or fabrication of the final restoration [1]. Typically, temporary cements, such as zinc oxide-based cements, are used for temporary cementation of provisional crowns [2]. However, it has been reported that when the remnants of temporary cement are not adequately removed from dentin surface, they can form a physical barrier, alter contact angle and dentin permeability [3,4], resulting in failure in hybrid layer formation and thereby decrease adhesion of the final restoration to dentin [2].

Mechanical removal of temporary cements only using hand instruments such as excavators is unfortunately not effective [4]. Although dentin surfaces may appear macroscopically clean, temporary cement residues could be observed microscopically on dentin surfaces that decrease the bonding of resin cements to dentin [4]. Thus, in order to effectively remove temporary cement residues and favor the longevity of adhesive restorations, cleaning agents for dentin such as chlorhexidine [5], ethylenediamine tetraacetic acid (EDTA) [6], polyacrylic acid [7] or mechanically cleaning using a mixture of pumice paste [8], prophylactic paste [9], air-abrasion using sodium bicarbonate spray [10] or air-abrasion with aluminum oxide particles (Al_2O_3) [7] have been proposed. Also, recently, a glycine-based powder for prophylaxis (Clinpro Prophy Powder, 3M ESPE, Irvine, CA, USA) has been suggested as an alternative to air-abrasion with sodium bicarbonate [11].

In general, cleaning methods used for removing temporary cement remnants from dentin surfaces could result in total or partial removal of the smear layer, thus facilitating the interaction between the resin cement and the dental substrate [7]. In addition, varying the application parameters of such agents can alter the results of the bond strength between the dentin/cement/zirconia interfaces [7]. Studies comparing the bond strength between dentin and resin cements after the use of different methods for temporary cement removal have presented diverse results [7,8,12]. The use of air-particle-abrasion with Al_2O_3 , pumice paste and 0.12% chlorhexidine before acid etching

did not significantly affect dentin bond strength, unlike the use of hydrogen peroxide and sodium bicarbonate jet, which significantly decreased the bonding [8]. Conversely, Santos et al. [7] reported higher bond strength values between resin cements and dentin when aluminum oxide air abrasion was used in comparison to mechanical cleaning with a hand instrument, pumice paste or sodium bicarbonate spray.

Although several studies have investigated the consequences of temporary cement removal methods from dentin on adhesion of resin cement, it is unclear as to which cleaning method is the more effective in removing the residues without compromising the bond strength of adhesively cemented zirconia restorations. Therefore, the objective of this study was to evaluate the effect of several cleaning methods employed for removing temporary cement on adhesion of adhesively luted zirconia to dentin and propose a clinical protocol. The hypothesis tested was that all cement residue removal techniques would not show significant difference in zirconia-dentin bond strength.

Materials and Methods

Tooth preparation

Forty non-carious human molars were disinfected (2% sodium hypochlorite solution for 2 h) and stored in distilled water at 23°C (ISO/TS 11405:2003). Each tooth was embedded in acrylic resin (JET Clássico, São Paulo, Brazil) using polyvinyl chloride (PVC) cylindrical tubes ($\varnothing=20$ mm; h=15 mm/1.2") until 3 mm below the cement-enamel junction. Each tooth was sectioned 3 mm below the occlusal surface using a diamond disc adapted in a cutting machine (Lab-Cut 1010, Extec, Enfield, NC, USA) under constant water irrigation in order to expose the superficial coronal dentin of 4 mm diameter (1.5-2 mm distant from the pulp). The surfaces were wet-ground finished with silicon carbide papers (#600) for 60 s using a polishing machine (Labpol 8-12, Extec, Enfield, NC, USA). The teeth were randomly divided into 5 groups (n=8) according to the factor "cleaning method" and stored in distilled

water at 23°C until further use in the study. This study was approved by the Research Ethics Committee of the Federal University of Paraíba (Protocol number: 435.230).

Preparing and cementing temporary crowns

The teeth were prepared with a chamfer finish line 2 mm below the occlusal surface with a width of 1 mm using diamond burs (#4138, KGSorensen, Barueri, SP, Brazil). Tooth preparations were made by one operator. Next, the teeth were isolated with vaseline and temporary crowns were made in acrylic resin (JET) (Figs. 1a-c). After the temporary crowns were polymerized and adjusted, the dentin surface was cleaned with a pumice paste (Maquira, Maringá, PR, Brazil), washed with an air-water jet for 30 s and dried with absorbent paper.

A thin layer of zinc oxide temporary cement without eugenol (RelyX Temp NE, 3M ESPE, Irvine, CA, USA) was applied on the intaglio surface of the temporary crown and positioned on each prepared tooth with slight pressure. The specimens (teeth with temporary crowns) were stored in distilled water at 37°C for 15 days.

Dentin cleaning methods

After storage in water, the temporary crowns were removed and the excess temporary cement was initially removed using an excavator (#5) (SSWhite Duflex, Juiz de Fora, MG, Brazil) in all groups, performed by a single operator. Next, the coronal dentin was cleaned from temporary cement residues according to one of the removal methods:

Air/water rinse (AW): Sprayed for 10 s at a distance of 5 mm.

Pumice paste (PP): The paste was applied using a brush for 10 s (Robson, Microdont, São Paulo, Brazil) in circular movements.

Aluminum oxide particles (AA): Air-particle abrasion with 50 µm Al₂O₃ (Bio-art, São Carlos, SP, Brazil) using a metallic device (Microjato Standard, Bioart, SP, Brazil) for 20 s, at 2.5 bar, at 90° inclination from a distance of 10 mm.

Sodium bicarbonate (SB): Air-particle abrasion with sodium bicarbonate (Biodinâmica, Paraná, PR, Brazil) with the same parameters in group AA using a chairside device (Jet Hand, Gnatus, Ribeirão Preto, SP, Brazil).

Glycine powder (CP): Air-particle abrasion with glycine powder (Clinpro Prophy Powder (3M ESPE, Irvine, CA, USA) under the same conditions as in group SB.

Adhesive cementation and aging

Zirconia blocks (Vita In-Ceram, YZ2000, VITA Zahnfabrik, Bad Säckingen, Germany) (20x19x15.5 mm³) were sectioned using a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, Illinois, USA) at slow speed (200 rpm) under water cooling to produce final specimens (5x15x20 mm³). The zirconia surfaces were polished using #600 and #1200 grit silicon carbide paper in sequence for 20 s. Next, a trephine bur was used perpendicularly to cut the ceramic specimens into cylinders (n=40) of 4.5 mm diameter with 5 mm height. The cylinders were cleaned in an ultrasonic (Cristófoli Biosafety Equipmen Ltda, Paraná, Brazil) with isopropyl alcohol for 5 minutes and then submitted to sintering (Zyrcomatt, Vita Zanhfabrik). Final dimensions (diameter: 3.4 mm; height: 4 mm) of the cylinders were verified with a digital caliper (Digital Paquimeter, Eccofer, São Paulo, Brazil) [13].

The zirconia cylinders were air-abraded with 50 µm Al₂O₃ for 20 s at 2.5 bar from a distance of 10 mm at an angle of 90° (Microjato Standard, Bioart, SP, Brazil). Next, the surfaces were cleaned in an ultrasonic cleaner (Cristófoli, Paraná, Brazil) with distilled water for 2 minutes and air-dried. A layer adhesive resin (Scotch Bond Universal, SBU, 3M ESP) was subsequently applied on the zirconia surfaces using a microbrush for 20 s and air-dried for 10 s.

After the cleaning procedures, the dentin surfaces were washed with air-water jet during 10 s and dried with absorbent paper. Next, the cementation area on the dentin surface was limited to 3.5 mm diameter by an adhesive tape. Then, SBU was applied for 20 s and air-dried for 10 s to remove the excess and to evaporate the solvent. Resin cement (RelyX Ultimate, 3M ESPE) was been handled according to the manufacturer's instructions and applied to the ceramic cylinders, positioned in the defined area on the dentin. Then a load of 750 g was applied

into the cylinders for 60 s. Cement excess was removed and photo-polymerized (1200mW/cm², Radii Cal, SDI, Bayswater, Victoria, Australia) for 20 s from each direction while the cylinder was under pressure and for a further 30 s without pressure. Finally, the specimens were stored in distilled water at 37°C for 90 days.

Shear bond strength test

After the aging period, the specimens were submitted shear bond strength test (SBS) (100 Kgf) in a universal testing machine (EMIC, model AG-X 10kN, Shimadzu, São José dos Pinhais, PR, Brazil). A chisel shaped device (Odeme Biotechnology/Luzerna, SC, Brazil) was coupled to the universal testing machine applying shear force into the ceramic-cement-dentin interface at a constant speed of 1 mm/min until failure. The bond strength was calculated using the formula: $R=F/A$, where: R=adhesive strength (MPa); F=force (N); and A=interfacial area (mm²). The cross-sectional area was 9.07 mm² [13].

Failure analysis

After debonding, the specimens surfaces were analyzed using a stereomicroscope (Nikon SMZ800, São Paulo, SP, Brazil) and the failure types were classified as: A1=adhesive at cement-ceramic interface; A2=adhesive at cement-dentin interface; C1=cohesive in cement; C2=cohesive in dentin; C3=cohesive in ceramic; A1C1=mixed (predominantly adhesive between cement and ceramic + cohesive in cement); A2C1=mixed (predominantly adhesive between cement and dentin + cohesive in cement).

Scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS)

Two specimens from each group were prepared for SEM (HITACHI, Model TM 3000, Tokyo, Japan) to be evaluated at x1000. On separate two specimens, EDS analysis (HITACHI, Model TM 3000, Tokyo, Japan) was performed on dentin surfaces after the studied cleaning methods.

Wettability measurements

On two additional specimens per cleaning method, contact angle was measured on the dentin surfaces [14]. Wettability was analyzed by the drop technique using an optical tensiometer (TL 1000, Theta Lite Attention,

Richfield, Staffordshire, UK). For this, three drops of distilled water were deposited on the dentin surface using a syringe (#1001 Gastight Syringes- 1 mL, Hamilton, Reno, NV, USA). After 5 s, images were made with a camera (Canon T3i, Canon Lens, Macro 100, Canon, São Paulo, Brazil) coupled at a fixed distance of 30 cm (Fig. 2). The mean contact angle was calculated using a software (Surftens V 4.5 s, OEG, Wildbahn 8i, Frankfurt, Germany) from three measurements per specimen.

Statistical analysis

Statistical analysis was performed using Statistix 8.0 Program for Windows (Analytical Software Inc, Tallahassee, FL, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, 1-way ANOVA and Tukey's tests were used where the bond strength was the dependent variable and cleaning methods (5 levels: PP, AA, SB, CP) as independent variables. EDS and wettability results were then qualitatively analyzed. The power of the sample was calculated using OpenEpi (Open Source Epidemiologic Statistics for Public Health, www.OpenEpi.com) site with the outcome of 84.41% for groups with eight specimens. P values less than 0.05 were considered to be statistically significant in all tests.

Results

Shear bond strength

ANOVA demonstrated that the SBS data were significantly affected by the tested cleaning methods ($p=0.02$). AA group (20.83 ± 7.8)^A presented significantly higher mean SBS than that of SB group (12.93 ± 3.1)^B and not to those of CP (14.30 ± 5.7)^{AB}, PP (13.58 ± 4.0)^{AB} and AW (13.32 ± 4.7)^{AB} (Fig. 3).

Failure types were predominantly A2C1 type (predominantly adhesive between cement and dentin + cohesive in cement) (60%) in AA, CP, PP and AW (Figs. 4a-b). The SB group presented 100% A1C1 type of failures (predominantly adhesive between cement and ceramic + cohesive in cement) (Figs. 5a-b).

SEM and EDS analysis

SEM images demonstrated temporary cement remnants in all groups after removal of temporary crowns and after cleaning methods. Less temporary cement residue was observed after cleaning with AW, SB, CP, and PP, AA (Figs. 6a-g). The presence of unobstructed dentinal tubules was also evident on the dentin surface after CP method (Fig. 6f) and it was possible to observe micro-retentions on the surface of the dentin in the specimens of the AA group (Fig. 6c).

The EDS analysis showed higher percentages of zinc in the specimens evaluated immediately after removal of the provisional crown (3.1%) and after the initial cleaning with the excavator (1.2%). AA group showed the presence of sodium (0.6%), alumina (1.8%) and zinc (0.1%). Zinc (0.2%) and sodium (0.8%) was also observed in the CP group while AW showed 0.1% Zn. The SB group presented Zinc (0.1%) and a higher percentage of sodium (2.7%) than those of other groups, indicating bicarbonate residues on the surface. The presence of sodium (0.9%) and the highest percentage of zinc (0.5%) was observed in the PP group.

Wettability

PP group ($100.1 \pm 6.9^\circ$) presented the highest mean contact angle, while the CP group the lowest value ($92.8 \pm 4.1^\circ$). Lower values show greater wettability on the dentin surface, which probably results in greater contact between the adhesive system and dentin, favoring adhesion. The other groups presented similar contact angle values (AW: 96.1 ± 9.9 ; AA: 97.2 ± 8.2 ; SB: 97.5 ± 10.9).

Discussion

This study evaluated the effect of different cleaning methods employed for removing temporary cement on adhesion of adhesively luted zirconia to dentin. According to the results of this study, since bond strength results were affected by the cleaning method the null hypothesis could be rejected.

The results showed that the removal of residual cement remnants by air-abrasion using $50 \mu\text{m Al}_2\text{O}_3$ particles promoted higher bond strength values of dentin-cement-zirconia assembly than with the sodium bicarbonate

group. Several previous studies reported favorable results for dentin cleaning using 50 μm Al_2O_3 particles where high bond strength values attributed to surface irregularities created on the dentin surface that have possibly increased the surface area available for adhesion [7,15,19]. SEM analysis of the dentin surfaces after AA in the present study showed the presence of these superficial irregularities that probably contributed to an increase in micromechanical retention of the adhesive and the resin cement on the dentin surface. Moreover, the adhesive system (SBU) used in this study presents phosphate monomers in its composition that also promotes chemical adhesion through its interaction with the metal oxides such as alumina. Thus, it can be anticipated that the interaction between the monomers and the alumina on the dentin surface after AA, as well as the adhesion to zirconia ceramic surface could have contributed to higher bond strength values in the AA group [19,20].

The bond strength results with the CP group was statistically similar to the AA group. Since CP is composed of pure glycine and has particle size in the range of 50 to 60 μm [21], which is similar to the aluminum oxide particle size used in this study (50 μm), suggesting CP promoted similar microretentions with that of AA. In a similar study, Frankenberger et al. [11] compared the effect of blasting with calcium carbonate and CP on the bond strength between human dentin and resin composite where cleaning agent promoted resulted in higher microtensile bond strength values. In addition, microscopically analysis revealed that the dentinal tubules were opened and they were not covered by a smear-layer after using this powder [11]. Corroborating these findings in the present study, it was also possible to observe unobliterated dentin tubules after the use of CP in SEM images which indicates that non-obliteration of the tubules allows greater contact of the adhesive system and the resin cement with the dentin surface, favoring adhesion. This method also presented the lowest mean contact angle values that indicates greater contact between the adhesive system and the dentine surface, that also contributed higher bond strength values.

One other commonly used cleaning method for dentin cleaning is mixing pumice paste and water. However, there are controversial reports in the literature even though it is frequently used. Some authors advocate the use

of PP for temporary cement cleaning from dentin surfaces while others report that this is not the most effective method [22]. The PP group herein, showed similar bond strength values and cement residues on the dentin surface to those found in the other groups, corroborating the results found by Soares et al. [8]. According to those authors, prophylaxis with PP and water results in a thinner smear-layer, thus facilitating acid etching in dentin during adhesive procedures [8]. In a similar study, Grasso et al. [15] reported that cleaning teeth with PP using rubber cup exhibited the least amount of temporary cement residue compared to other cleaning methods such as the use of excavator, AW or 0.12% chlorhexidine digluconate. However, PP also presented the best wettability and the highest percentage of zinc (0.5%) according to the EDS analysis when compared to others. In addition, the failure analysis revealed predominantly (60%) A2C1 failure, which suggests stronger adhesion between cement and ceramic when compared to adhesion between cement and dentin. These results indicate that this method can be used in order to promote an acceptable bond between dentin and resin cement, providing that it did not promote greater flow in the adhesive system or intimate contact with the dentin surface.

The AW group also showed statistically similar bond strength values with the other groups and presented less amount of temporary cement residues and lower wettability, indicating that this group is also a suitable method for removing temporary cement from the dentin surface. In a similar study, Atlintas et al. [23] evaluated the effect of three temporary cement (Cavex, Dycal, Tempond Clear) removal using cleaning methods with excavator #5, air/water rinse and optic cleaning on the bond strength between lithium disilicate ceramic and resin cement. The results verified higher bond strength values with excavator #5 and air/water rinse compared to cleaning with a brush. However, the failure analysis in this study revealed that 60% adhesives failures between cement and dentin and cohesive in cement. The reason for this according to Sarac et al. [5] was the presence of temporary cement residues which obliterated dentinal tubules, making penetration of the resin cement in the dentin difficult during adhesive procedures and yielding to weakest adhesion.

In this study, SB group demonstrated lower mean bond strength compared too AA group and was similar to the other groups, which indicates that bicarbonate residues can be incorporated into the hybrid layer and not interfere with adhesion when self-etching adhesives (SBU) are used [16]. Nishimura et al. [24] also evaluated the effect of sodium bicarbonate and crystalline cellulose on bond strength between human dentin and a self-etching adhesive system where blasting sodium bicarbonate particles decreased adhesion. The authors postulated that blasting sodium bicarbonate particles can cause physical and chemical changes in the collagen network, reducing the permeability to the adhesive system [7,21]. In addition, they consider that bicarbonate residues remain on the surface even after washing with water, causing an increase in pH, and thus altering the acidic monomer action of the evaluated self-etching adhesive as it cannot completely demineralize the dentin smear layer [7,21]. Failure analysis in this study, indicated 100% mixed failures in the SB group, being predominantly adhesive at the cement-ceramic interface along with cohesive failure in the cement. These findings imply good interaction between the adhesive system, resin cement and the dentin. Nevertheless, further research evaluating the interaction of SB with self-etching adhesive systems is necessary in order to confirm the results.

Based on the SEM images and EDS analysis, the null hypothesis could be partially accepted. SEM images and EDS analysis qualitatively denoted few remaining temporary cement particles on the dentin surfaces of all groups. Considering the percentage of zinc, which is a component of the temporary cement, it was possible to observe higher percentages on the dentin specimens after removal of the provisional crown (3.1%) and after cleaning with excavator (1.2%) when compared to the other evaluated cleaning methods: AA (0.1%); CP (0.2%); AW (0.1%); SB (0.1%) and PP (0.5%). These findings signify that all cleaning methods removed the temporary cement residues, but not completely.

Regarding the clinical application of the temporary cement debris removal methods used in this study, it is important to consider that AA resulted in cracks in the dentin surface that can alter its structural interference and hardness. Aluminum oxide particles and dentin fragments found detached after AA was reported to decrease

adhesion [25]. Some requirements for this method are also necessary such as high potency suction and rubber-dam isolation. On the contrary, the clinical use of CP has some advantages when compared to SB in that CP presents a less abrasive effect due to smaller-sized particles [26] and has no unpleasant salty taste as it is based on glycine [27]. It also results in less gingival erosion and can be used without rubber-dam isolation [27]. The main results of different cleaning methods of the studies reported in the literature are summarized in Table 1. Further research involving more aggressive aging protocols, other resin cements, as well as randomized controlled clinical trials should be developed to complement and validate the results obtained in the present study.

Conclusions

From this study, the following could be concluded:

- Temporary cement residues could be cleaned effectively from dentin surfaces using air-water rinse, pumice paste, sodium bicarbonate, glycine powder or air-abrasion with aluminum oxide particles where the latter resulted in the highest mean bond strength which was significantly higher than with that of sodium bicarbonate spray.
- After cleaning with pumice paste, the highest (less wettability) and with glycine powder the lowest contact angle (better wettability) values were obtained.
- Failure types were predominantly mixed type of failures either between dentin-cement or cement-zirconia in all groups.

Clinical Relevance

Temporary cement residues could be cleaned effectively from dentin surfaces where air-abrasion with 50 μ m aluminum oxide particles showed a more promising mean bond strength value than with bicarbonate air-abrasion when zirconia was luted with the tested resin cement and the adhesive resin on dentin.

Conflict of interest

No potential conflict of interest was reported by the authors.

References

- 1] Abdulmohsen B, Parker S, Braden M, et al. A study to investigate and compare the physicommechanical properties of experimental and commercial temporary crown and bridge materials. *Dent. Mater.* 2016;32:200-210.
- 2] Yap AUJ, Shah KC, Loh ET, et al. Influence of Eugenol-Containing Temporary Restorations on Bond Strength of Composite to Dentin. *Oper.Dent.* 2001;26:556-561.
- 3] Terata R, Nakashima K, Obara M, et al. Characterization of enamel and dentin surfaces after removal of temporary cement-effect of temporary cement on tensile bond strength of resin luting cement. *Dent. Mater. J.* 1994;13:148-154.
- 4] Watanabe EK, Yatani H, Ishikawa K, et al. Pilot study of conditioner/primer effects on resin–dentin bonding after provisional cement contamination using SEM, energy dispersive X-ray spectroscopy, and bond strength evaluation measures. *J. Prosthet. Dent.* 2000;83:349-55.
- 5] Sarac D, Bulucu B, Sarac YS, et al. The effect of dentin-cleaning agents on resin cement bond strength to dentin. *J. Am. Dent. Assoc.* 2008;139:751-758.
- 6] Munirathinam D, Mohanaj D, Beganam M. Efficacy of various cleansing techniques on dentin wettability and its influence on shear bond strength of a resin luting agent. *J. Adv. Prosthodont.* 2012;4:139-145.
- 7] Santos MJMC, Bapoo H, Rizkalla AS, et al. Effect of dentin-cleaning techniques on the shear bond strength of self adhesive resin luting cement to dentin. *Oper. Dent.* 2011;36:512-520.
- 8] Soares CJ, Pereira JC, Souza SJB, et al: The Effect of prophylaxis method on microtensile bond strength of indirect restorations to dentin. *Oper. Dent.* 2012;37:602-609.
- 9] Özcan M, Lamperti S. Effect of mechanical and air-particle cleansing protocols of provisional cement on immediate dentin sealing layer and subsequent adhesion of resin composite cement. *J. Adhes. Sci. Technol.* 2015;29:2731-2743.

- 10] Sol E, Espasa E, Boj JR, et al. Effect of different prophylaxis methods on sealant adhesion. *J. Clin. Pediatr. Dent.* 2000;24:211-214.
- 11] Frankenberger R, Lohbauer U, Tay FR, et al. The effect of different air-polishing powders on dentin bonding. *Adhes Dent.* 2007;9:381-9.
- 12] Rosin C, Arana-Chavez VE, Garone Netto N, et al: Effects of cleaning agents on bond strength to dentin. *Braz. Oral Res.* 2005;19:127-133.
- 13] Bottino MA, Bergoli C, Lima EG, et al. Bonding of Y-TZP to dentin: Effects of Y-TZP surface conditioning, resin cement type, and aging. *Oper. Dent.* 2014;39:291-300.
- 14] Good RJ, Contact-Angle, wetting, and adhesion - A critical review. *J. Adhes. Sci. Technol.* 1992;6:1269-1302.
- 15] Grasso CA, Caluori DM, Goldstein GR, et al. In vivo evaluation of three cleansing techniques for prepared abutment teeth. *J. Prosthet. Dent.* 2002;88:437.
- 16] Chaiyabutr Y, Kois JC. The effects of tooth preparation cleansing protocols on the bond strength of self-adhesive resin luting cement to contaminated dentin. *Oper. Dent.* 2008;33:556-563.
- 17] Otani A, Amaral M, May LG, et al. A critical evaluation of Bond strength tests for the assessment of bonding to Y-TZP. *Dent. Mater.* 2015;31:648-656.
- 18] Della Bona, A. Bonding to ceramics: scientific evidences for clinical dentistry. (ed): São Paulo: Artes Médicas Ltd., ed. 1 (Chapter 4), 2009.
- 19] Craciunescu E, Sinescu C, Negrutiu ML, et al. Shear bond strength tests of zirconia veneering ceramics after chipping repair. *J. Adhes. Sci. Technol.* 2016;30:666-676.
- 20] Amaral M, Belli R, Cesar PF, et al. The potential of novel primers and universal adhesives to bond to zirconia. *J. Dent.* 2014;42:90-98.

- 21] Schwarz F, Ferrari D, Popovski K, et al: Influence of different air-abrasive powders on cell viability at biologically contaminated titanium dental implants surfaces. *J. Biomed. Mater. Res. B Appl. Biomater.* 2009;88:83-11.
- 22] Gultz J, Kaim V, Scherer W. Treating enamel surfaces with a prepared pumice prophylaxis paste prior to bonding. *Gen. Dent.* 1999;47:200-201.
- 23] Altintas SH, Tak O, Secilmis A, et al. Effect of provisional cements on shear bond strength of porcelain laminate veneers. *Eur. J. Dent.* 2011;5:373-379.
- 24] Nishimura K, Nikaido T, Foxton RM, et al. Effect of Air-powder polishing on dentin adhesion of a self-etching primer bonding system. *Dent. Mater. J.* 2005;24:59-65.
- 25] Fonseca RB, Martins LR, Quagliatto OS, et al. Influence of provisional cements on ultimate bond strength of indirect composite restorations to dentin. *J. Adhes. Dent.* 2005;7:225-230.
- 26] Pelka MA, Altmaier K, Petschelt A, et al. The effect of air-polishing abrasives on wear of direct restoration materials and sealants. *J. Am. Dent. Assoc.* 2010;141:63-70.
- 27] Petersilka GJ, Steinmann D, Häberlein I, et al. Subgingival plaque removal in buccal and lingual sites using a novel low abrasive air-polishing powder. *J. Clin. Periodontol.* 2003;30:328-333.

Captions to figures and tables:

Tables:

Table 1. Summary of previous studies in the literature on the topic of cleaning temporary cements from dentin surfaces and the main outcomes.

Figures:

figs. 1a-c **a)** Chamfer finish line prepared on the occlusal surface with a width of 1 mm, **b)** Profile view of the chamfer finish line prepared, **c)** Temporary crown made of acrylic resin cemented on dentin.

figs. 2a-c Drops of distilled water on the dentin surface after cleaning methods to measure the contact angle, **a)** one drop, **b)** two drops, **c)** three drops.

fig. 3 Mean shear bond strength (MPa) and standard deviations (SD) of the experimental groups. *significant statistical difference (Tukey`s test, 5%). Same superscript letters indicate no significant difference. AW: air-water abrasive (AW); PP: pumice paste; AA: air-abrasion with 50 µm aluminum oxide particles (Al₂O₃); SB: sodium bicarbonate spray; CP: glycine powder.

figs. 4 a-b Representative photos of optical microscope images (x40) of mixed failure type A2C1 (predominantly adhesive between cement and dentin + cohesive in cement), **a)** cement-dentin interface (arrow: dentin); **b)** cement-ceramic interface (asterisk: cement resin; radius: ceramic).

figs. 5a-b Representative photos of optical microscope images (x40) of mixed failure type A1C1 (predominantly adhesive between cement and ceramic + cohesive in cement), **a)** cement-dentin interface; (asterisk: cement resin, arrow: dentin), **b)** cement-ceramic interface (asterisk: cement resin, radius: ceramic, arrow: dentin).

figs. 6a-g SEM images (x1000) of dentin surfaces **a)** after removal of the provisional cement, **b)** cleaning with excavator, **c)** AA, **d)** AW, **e)** SB, **f)** CP, **g)** PP. Arrow: temporary cement, Radius: coronal dentin, Star: superficial irregularity, Wind rose: dentinal tubules. See Fig. 3 for group abbreviations.

Tables:

Summary of Studies in the Literature			Results of our study	
Author/Year	Cleaning Methods	Main Outcome	Cleaning Methods investigated	Main results
Chaiyabutr et al., 2008	1) Hand instrument (excavator) 2) Propy with a mixture of flour pumice and water 3) Aluminous oxide abrasion (particle size of 27 µm at 40 psi) 4) Aluminous oxide abrasion (particle size of 50 µm at 40 psi)	Aluminous oxide particle provided highest values of bond strength, while hand instrument excava was the lowest ($p<0.05$)		
Grasso et al., 2002	1) Hand instrument (dental explorer) 2) Air-water spray 3) Propy cup with fine flour pumice 4) Cotton pellet soaked in 0.12% chlorhexidine gluconate	Pumice cleansing technique was significantly better than the explorer or the cotton pellet/chlorhexidine gluconate technique		
Frankenberger et al., 2007	1) Prophypearls (calcium carbonate) 2) ClinPro Prophypowder (glycine)	Calcium carbonate air polishing generally caused significantly reduced dentin bond strengths ($p<0.05$). Glycine did not affect dentin bonding performance	<ul style="list-style-type: none"> • Air/water rinse • Pumice paste • Air-abrasion with 50 µm Al₂O₃ • Air-abrasion with sodium bicarbonate • Air-abrasion with Clinpro Powder 	All evaluated methods were effective regarding the removal of temporary cement residues. Air-abrasion with 50 µm aluminum oxide particles promoted higher mean bond strength between zirconia and dentin than sodium bicarbonate spray.
Altintas et al., 2011	1) Dental explorer 2) Air-water spray 3) Cleaning bur (Opticlean)	In all groups, explorer and air-water spray was more effective in removal of residual provisional cement than with a cleaning bur ($p<0.05$)		
Saraç et al., 2008	Cleaning agent: 1) Sikko Tim (contains ethanol, ethyl acetate and acetone-based disinfectant) 2) Cavity Cleanser (2% chlorhexidine digluconate) 3) Consepsis Scrub (2% chlorhexidine digluconate and glass)	Sikko Tim and Consepsis Scrub were effective in removing provisional cement ($\alpha=.05$)		

	particles).	
Nishimura et al., 2005	1) Air-powder polishing with sodium bicarbonate (SB) 2) Crystalline cellulose (CC)	Air-powder polishing with SB affected bond strength to dentin, while that with CC did not influence bond strength ($p < 0.05$)
Rosin et al., 2005	1) Sodium bicarbonate jet 2) Pumice paste plus 3) A biologic detergent (Tergestesim) 4) Air water spray	No statistical differences among the cleaning agents and neither between their interactions with the bonding systems ($p < 0.001$)
Özcan et al., 2015	1) Air-borne particle abrasion with 50- μm Al_2O_3 particles at 2 bar (AL2) 2) Air-borne particle abrasion with 50- μm Al_2O_3 particles at 3.5 bar (AL3.5) 3) Air-borne particle abrasion with 30 μm SiO_2 particles at 2 bar (SL2) 4) Air-borne particle abrasion with 30- μm SiO_2 particles at 3.5 bar (SL3.5) 5) Prophylaxy paste (Cleanic) 6) Pumice-water slurry at 1500 rpm for 15 s	Air-abrasion with 50 μm Al_2O_3 parti and mechanical cleansing method: prophylaxy paste or pumice-water indicating the need for some press remove the provisional cement from surfaces ($\alpha = 0.05$)
Munirathinam et al., 2012	1) Control-air-water spray 2) Pumice prophylaxis, 3) Ultrasonic scaler with 0.2% Chlorhexidine gluconate 4) 17% EDTA	EDTA was the most effective dentin cleaning agent among the compared groups ($\alpha = 0.05$)
Fonseca et al., 2005	1) Hand scaler for 10 s 2) Pumice-water slurry for 10 s 3) aluminum oxide sandblasting for 10 s	Aluminum oxide sandblasting provided the highest values of bond strength and calcium hydroxide the lowest ($p < 0.05$)
Santos et al., 2011	1) Excavator (control) 2) 0.12% chlorhexidine digluconate 3) 40% polyacrylic acid 4) Mixture of flour pumice and water	Sandblasting with aluminum oxide produced significantly higher shear bond strength values compared with any other treatment group ($p < 0.05$)

5) Sandblasting with
50 μ m aluminum oxide particles

Table 1. Summary of previous studies in the literature on the topic of cleaning temporary cements from dentin surfaces and the main outcomes.

Figures:

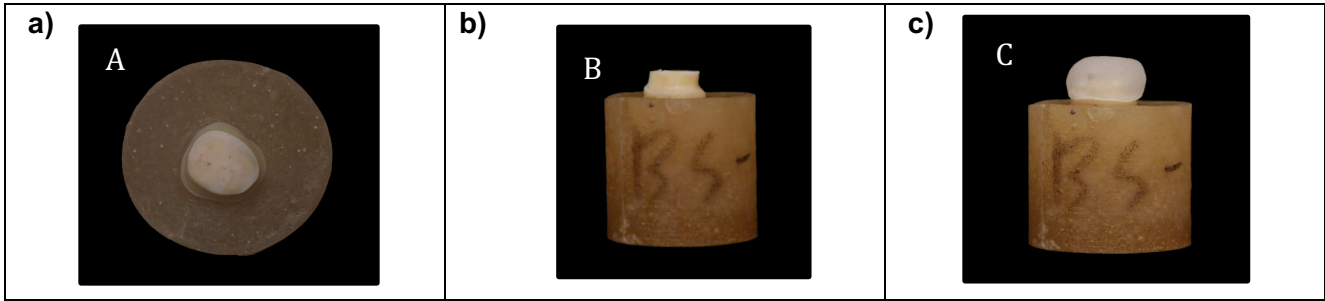


Fig. 1a-c a) Chamfer finish line prepared on the occlusal surface with a width of 1 mm, b) Profile view of the chamfer finish line prepared, c) Temporary crown made of acrylic resin cemented on dentin.

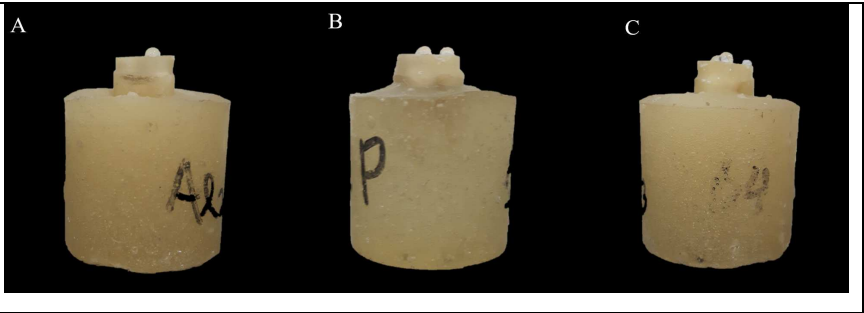


Fig. 2a-c Drops of distilled water on the dentin surface after cleaning methods to measure the contact angle, a) one drop, b) two drops, c) three drops.

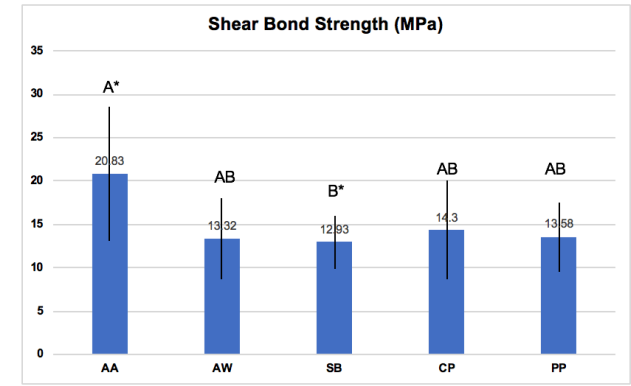
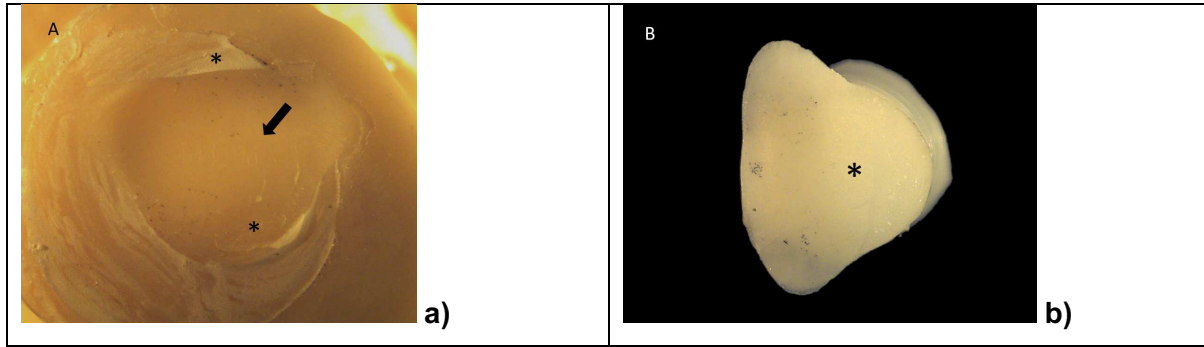
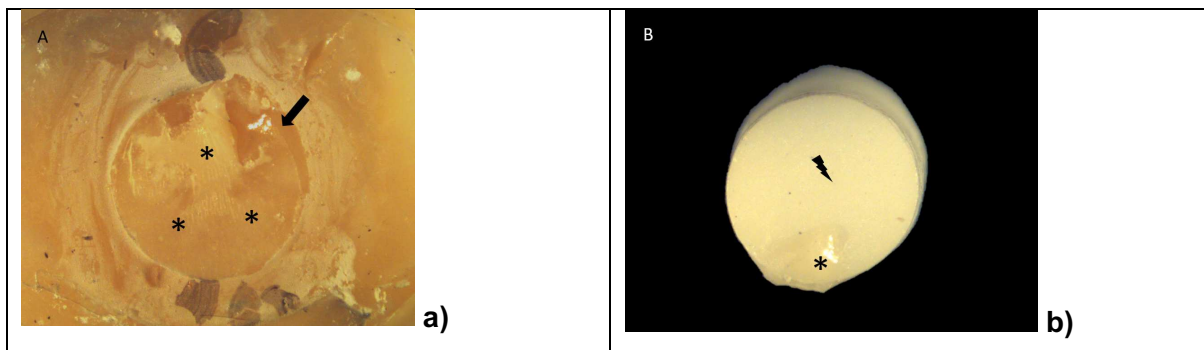


Fig. 3 Mean shear bond strength (MPa) and standard deviations (SD) of the experimental groups. *significant statistical difference (Tukey's test, 5%). Same superscript letters indicate no significant difference. AW: air-water rinse (AW); PP: pumice paste; AA: air-abrasion with 50 μ m aluminum oxide particles (Al_2O_3); SB: sodium bicarbonate spray; CP: glycine powder.



figs. 4 a-b Representative photos of optical microscope images (x40) of mixed failure type A2C1 (predominantly adhesive between cement and dentin + cohesive in cement), **a)** cement-dentin interface (arrow: dentin); **b)** cement-ceramic interface (asterisk: cement resin; radius: ceramic).



figs. 5a-b Representative photos of optical microscope images (x40) of mixed failure type A1C1 (predominantly adhesive between cement and ceramic + cohesive in cement), **a)** cement-dentin interface; (asterisk: cement resin, arrow: dentin), **b)** cement-ceramic interface (asterisk: cement resin, radius: ceramic, arrow: dentin).

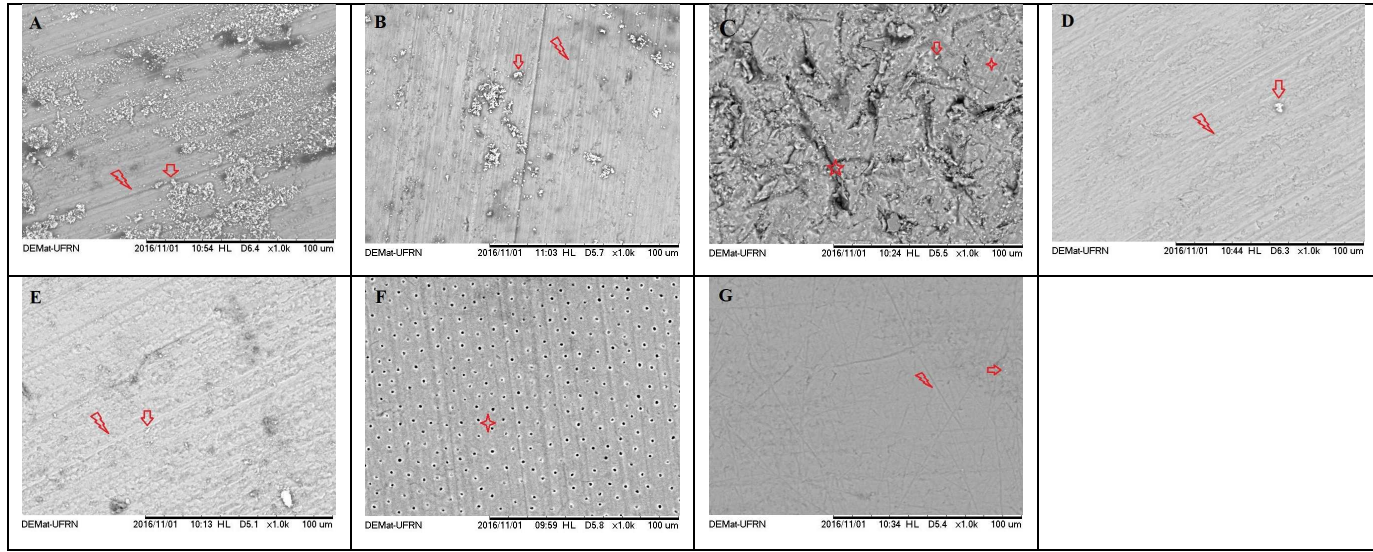


Fig. 6a-g SEM images (x1000) of dentin surfaces **a)** after removal of the provisional cement, **b)** cleaning with excavator, **c)** AW, **d)** SB, **e)** CP, **f)** PP, **g)** PP. Arrow: temporary cement, Radius: coronal dentin, Star: superficial irregularity, Wind rose: dentinal tubules. See Fig. 3 for group abbreviations.